

## Office of Naval Research International Field Office

### 30. Nanostructured Al-Fe Alloys

Dr. Jun Kameda

July 17, 2003

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These reports summarize global activities of S&T Associate Directors of  
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#### Contents:

1. Summary
2. Background
3. Assessment
4. Points of Contact

***Key Words: Nanostructured Al-Fe alloys, Strain rate effect, Hopkinson-bar tests, Electron beam deposition method, Super-saturated solid solution***

## **1. Summary**

Prof. Mukai has expertise in studying superplasticity and dynamic deformation behavior in nanostructured lightweight materials. He was an invited presenter at the TMS-Spring Meeting in San Diego CA, visited Boston MA and Washington DC under the Visitor Support Program (VSP) of the ONRIFO on March 2-14, 2003. One of his VSP objectives is to seek U.S. collaborators for a NICOP proposal. He has presented his work on nanostructured Al and Mg alloys to Dr. Vasudevan, Scientific Officer of Materials S & T Division (ONR 332). He has maintained collaborative efforts with Prof. Suresh since he spent a year at MIT as a visiting scientist. This report contains his presentation summary.

## **2. Background**

Nanostructured metallic and ceramic materials have unique mechanical properties. Currently numerous programs attempt to tailor advanced structural and functional nanostructured materials. Mukai's group has developed high strength (650-850 MPa) Al alloys by applying an electron beam deposition (EBD) method to achieve super-saturated solid solution of Fe, leading to the formation of nano-sized grains. The nanostructured Al-Fe alloys show stronger dependence of strength on the Fe content and weaker dependence on the strain rate, compared to other ultra-fine grained Al-Fe alloys.

## **3. Assessment**

The application of the EBD method enables to fabricate high strength Al-base alloys. While the applicability of such alloys is restricted in functional components due to the size limitation, such a study will provoke scientific interests.

#### 4. Points of Contact

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*Information  
@ONR Headquarter  
March 12, 2003*

# Controlling grain size and texture to develop a tough solid solution nano- crystalline aluminum alloy

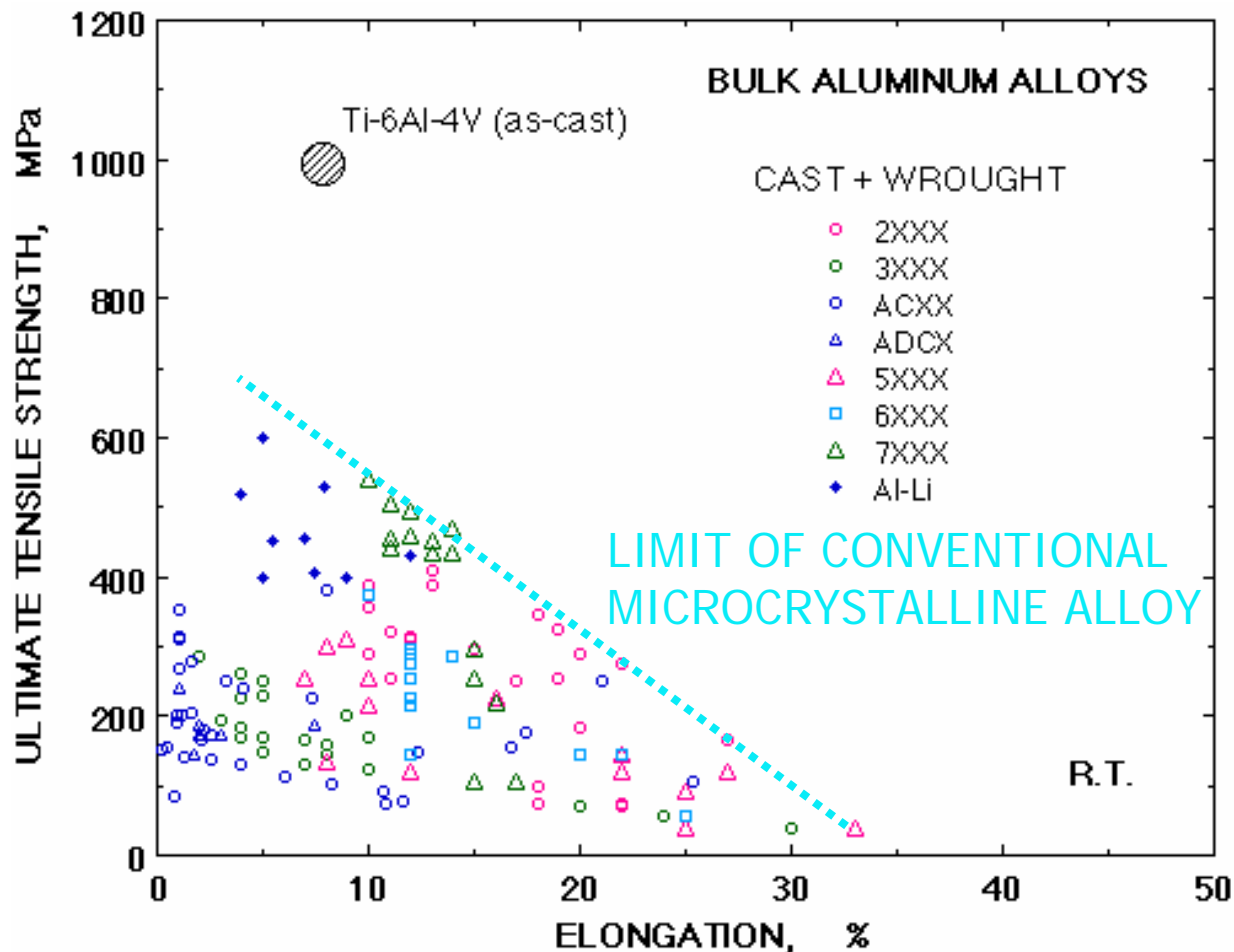
Toshiji Mukai

*Research Center for Advanced Science and Technology,  
The University of Tokyo, JAPAN*

# Motivation

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RCAST,  
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- Replace Ti alloys to Al alloys for the weight reduction(- 40%) in future Aerospace Application.
- However, conventional Al alloys does not show sufficient strength and ductility balance.



# Previous Works of Grain Refinement in Bulk Aluminum

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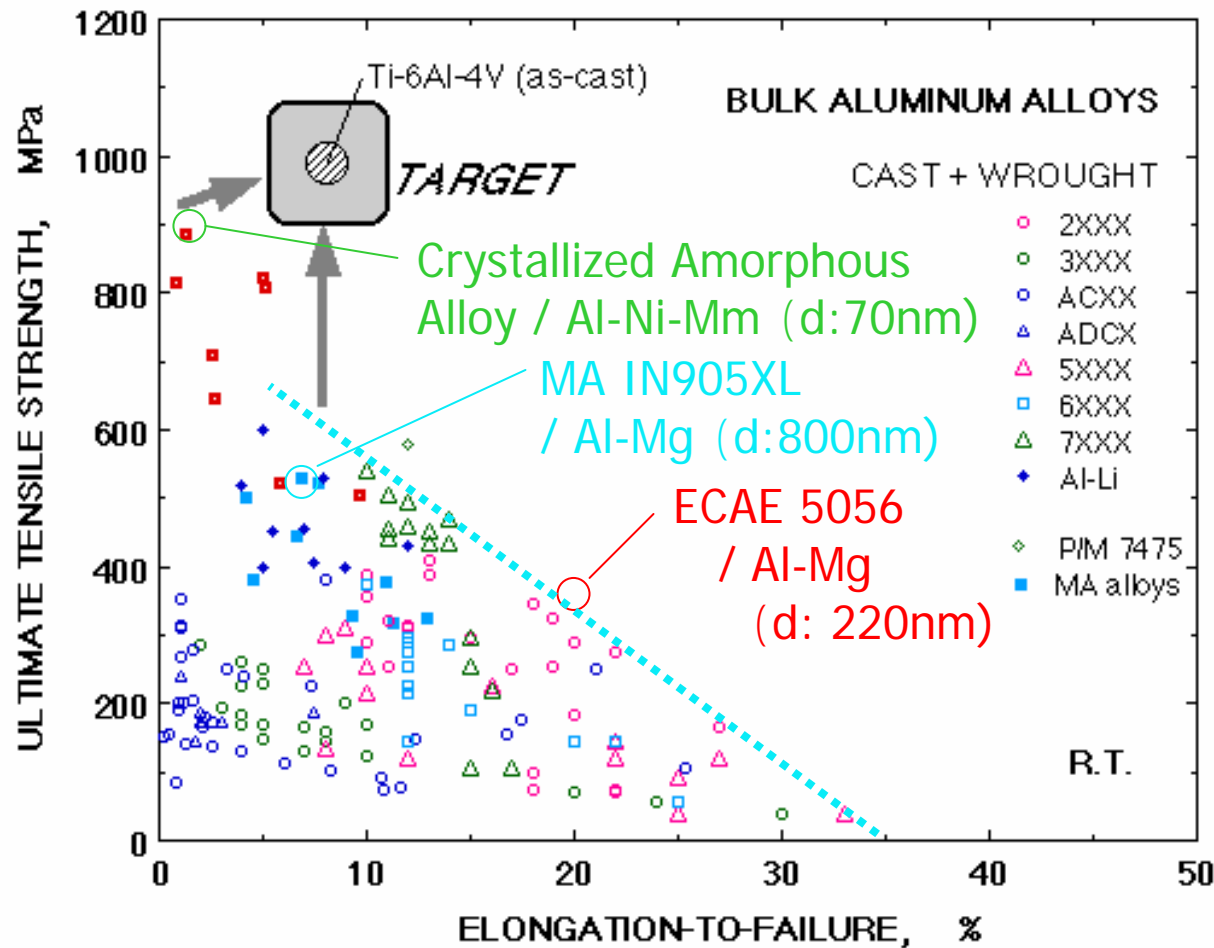
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## ❑ Developed by Advanced Technique

- Powder Metallurgy(P/M)
- Mechanical Alloying(MA) +P/M  
[Al-Mg-Li,  $d \sim 800\text{nm}$ ]
- Crystallization of Amorphous Powder +P/M  
[Al-Ni-Mm,  $d \sim 70\text{ nm}$ ]
- Consolidation of Nano-crystalline Powder +P/M  
[Al-Ni-Mm-Zr,  $d \sim 100\text{ nm}$ ]
- Physical Vapor Deposition(PVD)
- Severe Plastic Deformation
  - Torsion Straining
  - Equal-Channel-Angular-Extrusion(ECAE)  
[Al-Mg-Mm,  $d \sim 220\text{ nm}$ ]

# Strength-Ductility Balance in Advanced Fine-Grained Al Alloys

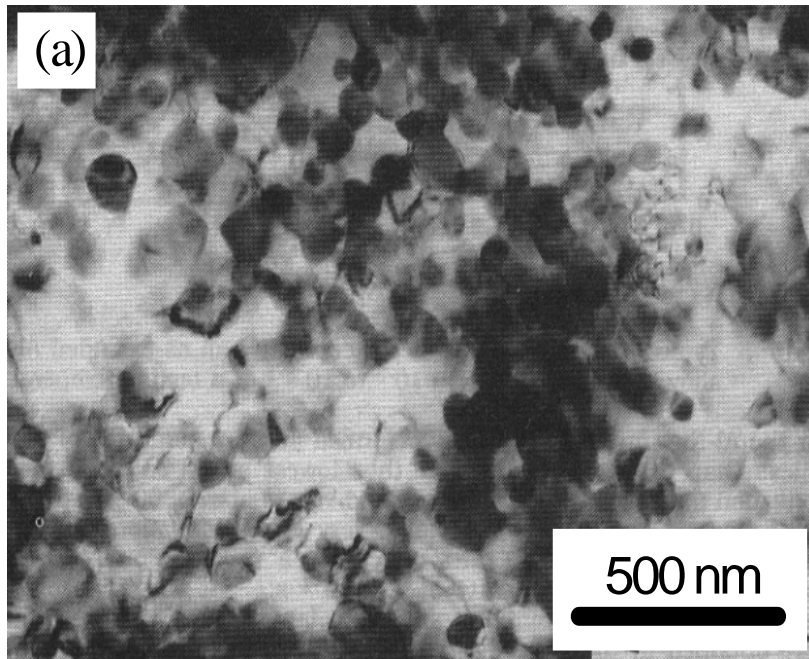
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RCAST,  
Univ. Tokyo



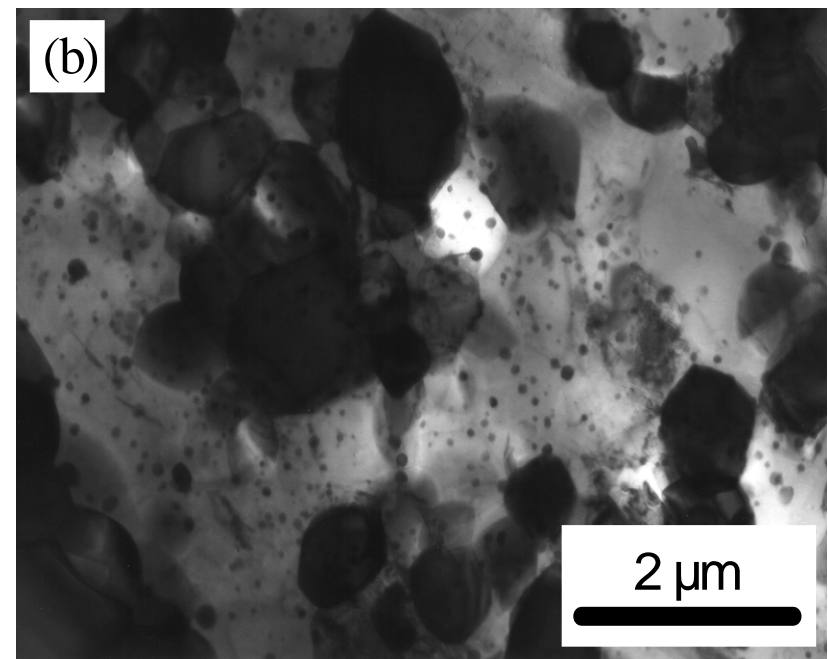
# Typical Microstructure of NC alloy crystallized from Amorphous Powder

[Al-14wt.% Ni-14 wt.% Mm, Crystallized from Amorphous]

(Supplied by YKK Co., Japan)



[As-extruded]



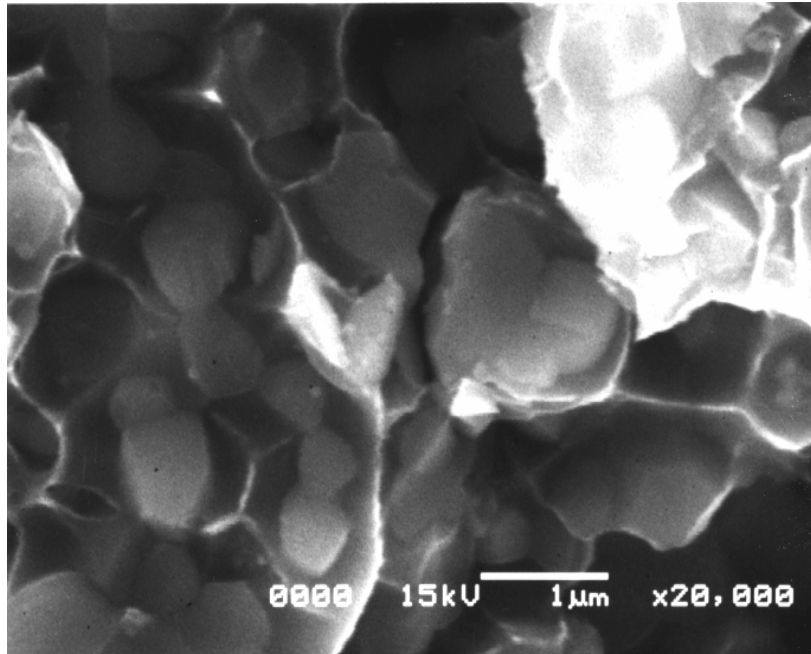
[Annealed at 773 K]



# Fracture Feature

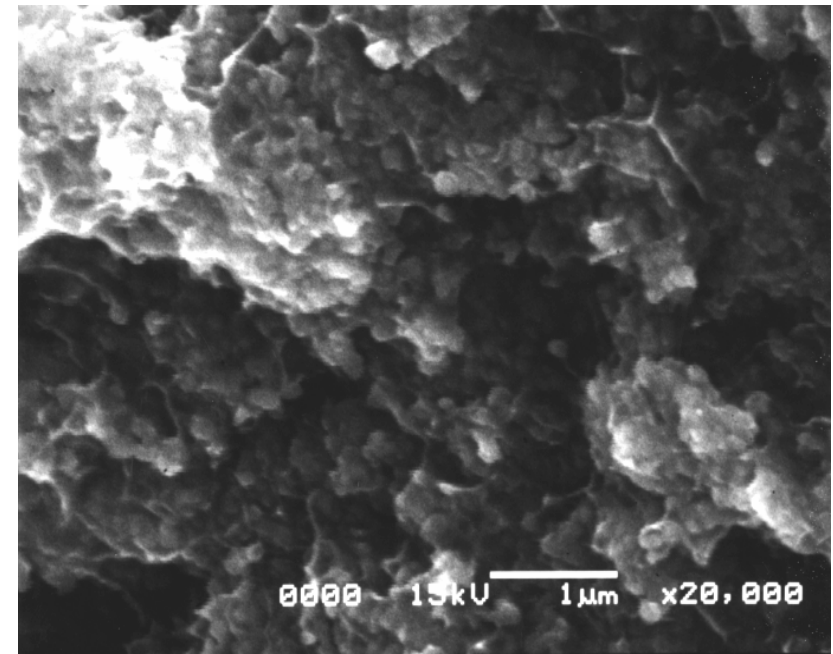
- Al-Ni-Mm Alloy (Crystallized from amorphous powder)
- At dynamic strain rate ( $10^3 \text{ s}^{-1}$ )

$[d = 1 \mu\text{m}]$



$[Elongation-to-Failure: 0.6\%]$

$[d = 70 \text{ nm}]$



$[Elongation-to-Failure: 1.2\%]$

- Brittle Fracture --- at the interface of Matrix/Second Phase
- Elongation-to-Failure increases with Grain Refinement

[Mukai, Higashi: Scripta mater. (2000)]

# Typical Microstructure of SMC alloy through ECAE

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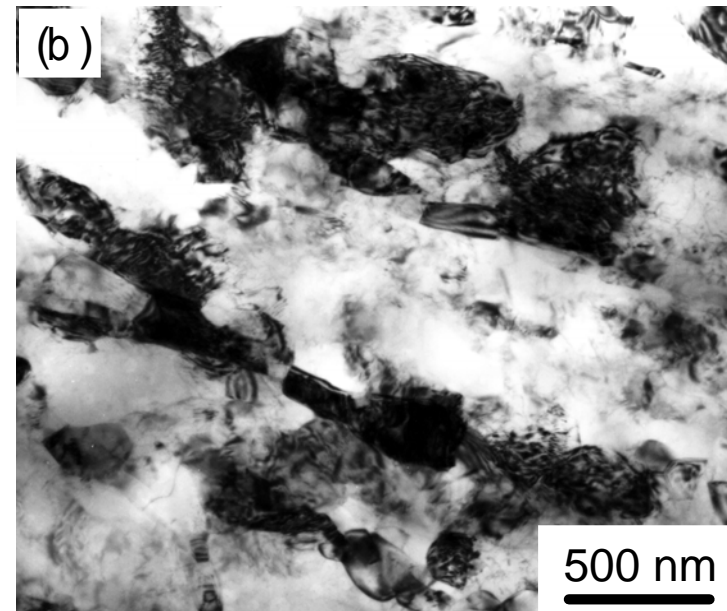
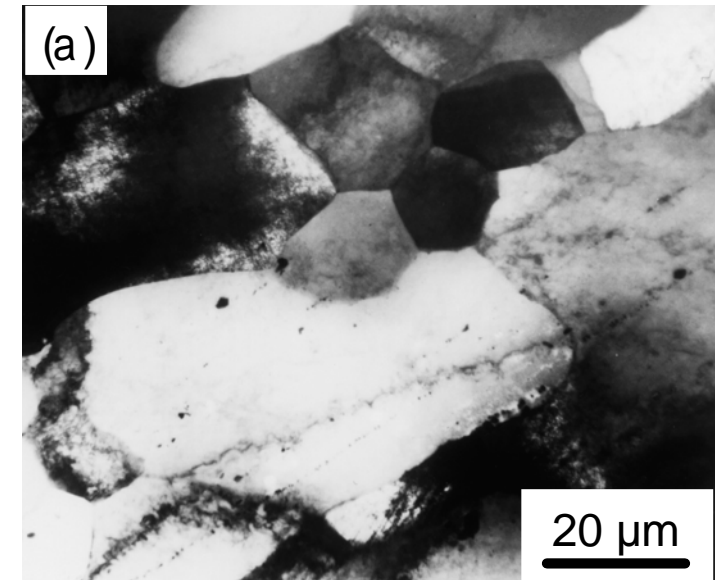
- Material: 5056 Al alloy  
[Al-4.8Mg-0.07Mn-0.06Cr-0.1Fe-0.06Si, wt.%]
- Grain Refinement:  
ECAE (Equal-Channel-Angular-Extrusion) Process

(a) Fully Annealed [ $d = 35 \mu\text{m}$ ]

(b) ECAE Alloy [ $l = 220 \text{ nm}$ ]

(c) ECAE + Annealed [ $l = 1 \mu\text{m}$ ]

(d) ECAE + Annealed [ $d = 10 \mu\text{m}$ ]



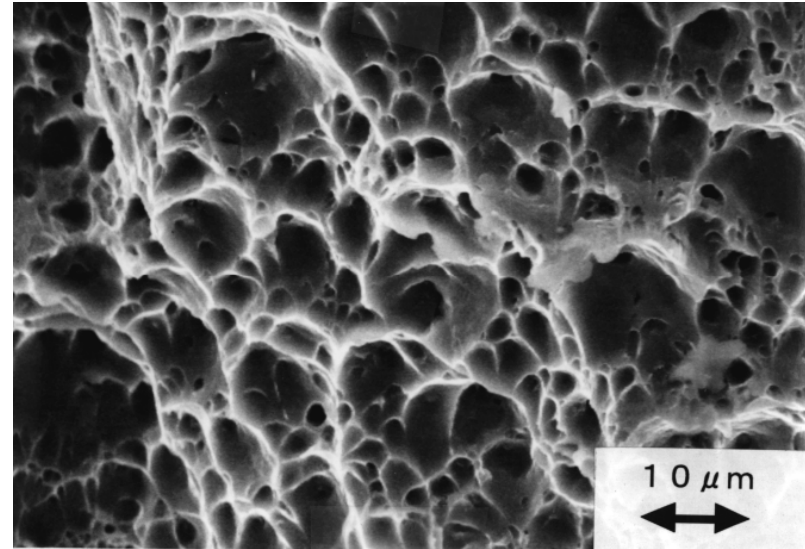
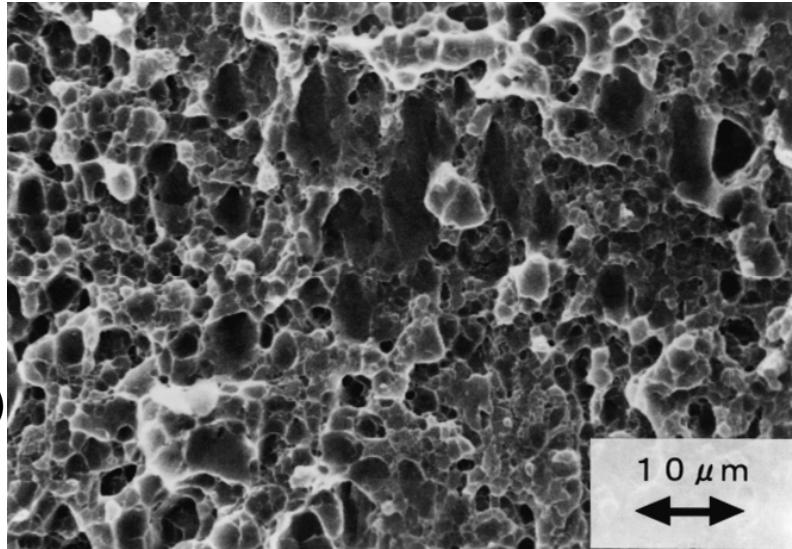
# Fracture Feature

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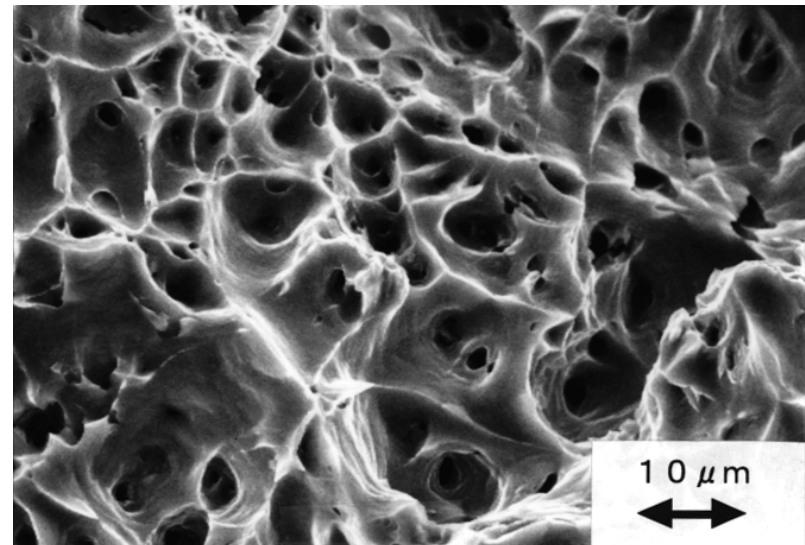
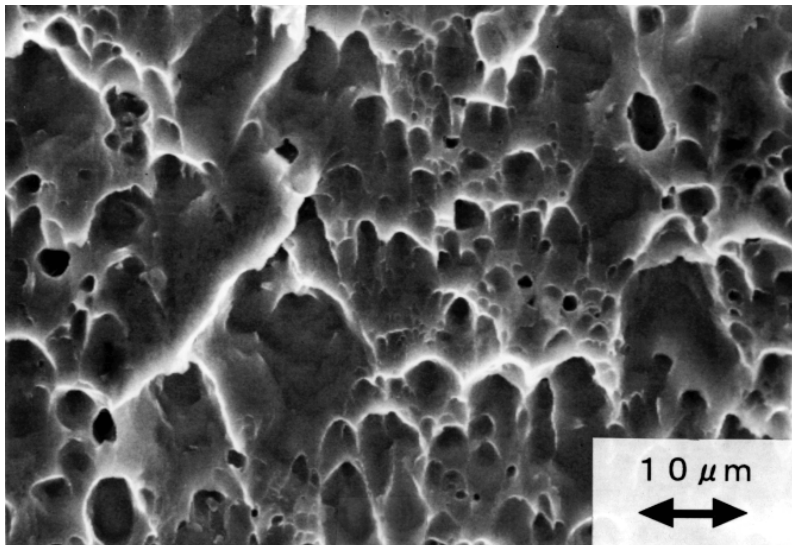
Q uasi-S tatic ( $10^{-3} \text{ s}^{-1}$ )

D ynam ic ( $10^3 \text{ s}^{-1}$ )

5056-  
E C A E  
( $0.22 \mu\text{m}$ )

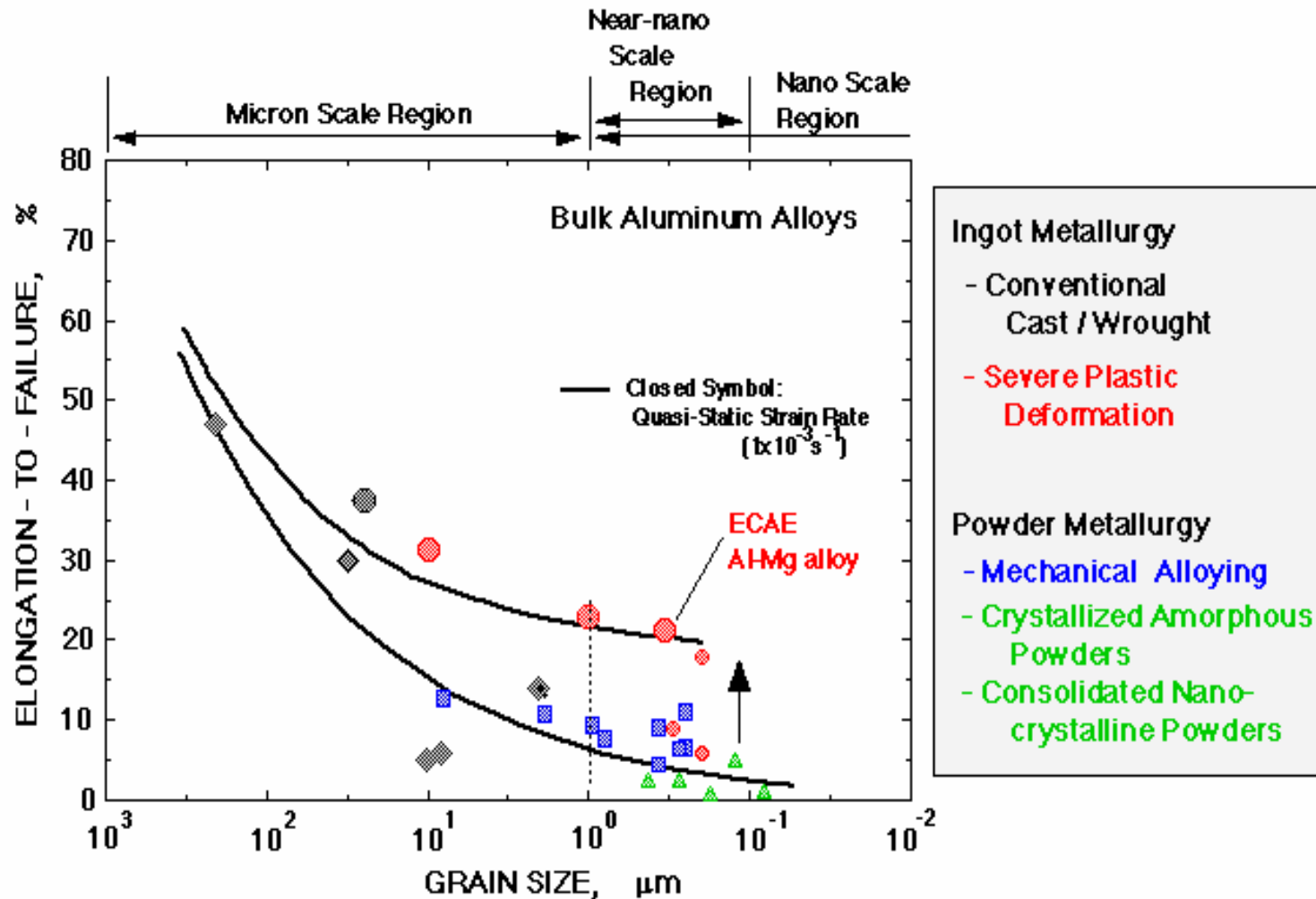


5056-0  
( $35 \mu\text{m}$ )



# Variation in Elongation with Refining Grain Size for Bulk Aluminum Alloys

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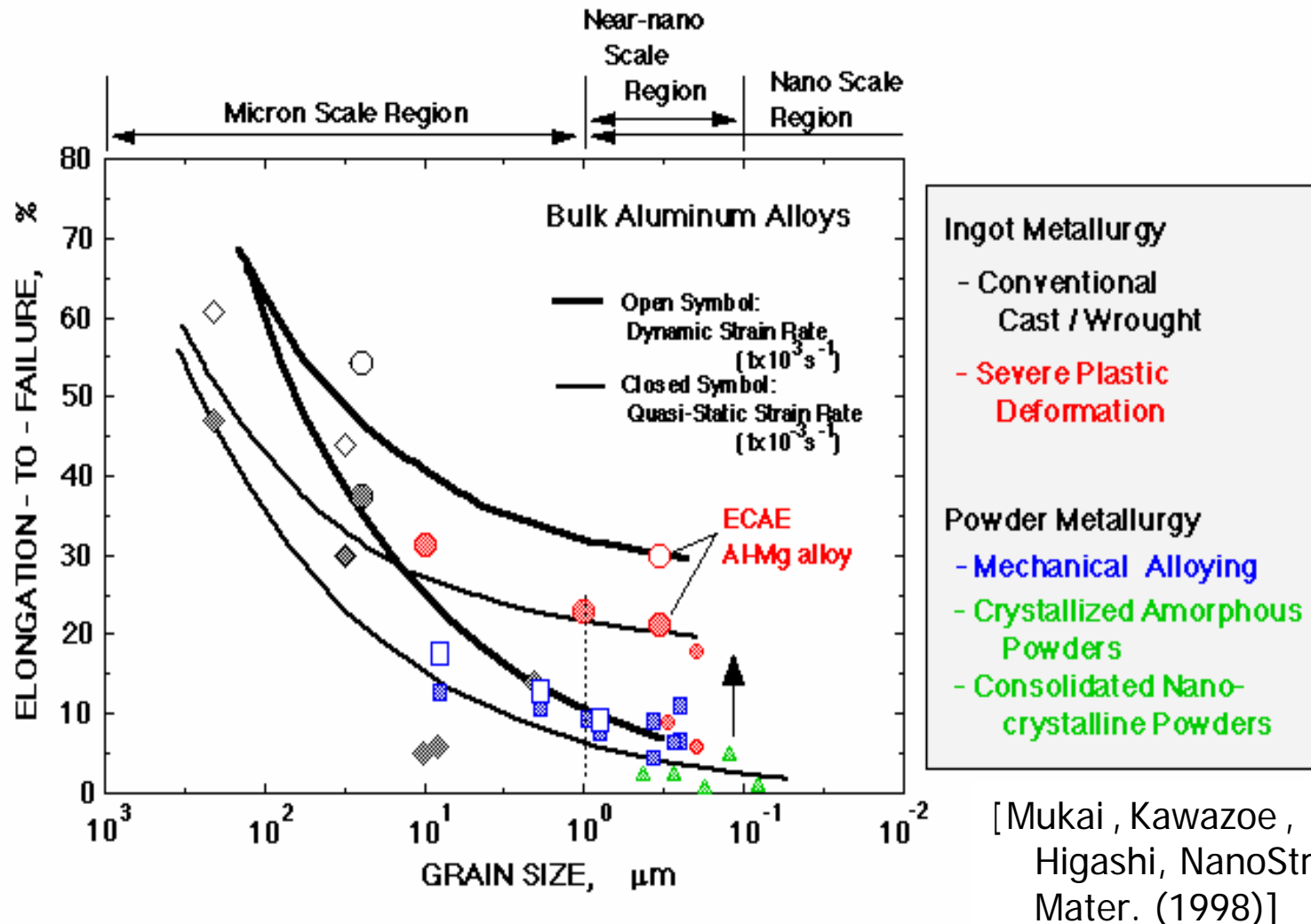


- Possibility of nano-crystalline/solid solution alloys



# Variation in Elongation with Refining Grain Size for Bulk Aluminum Alloys

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Univ. Tokyo



- Possibility of nano-crystalline/solid solution alloys

# Purpose of this study

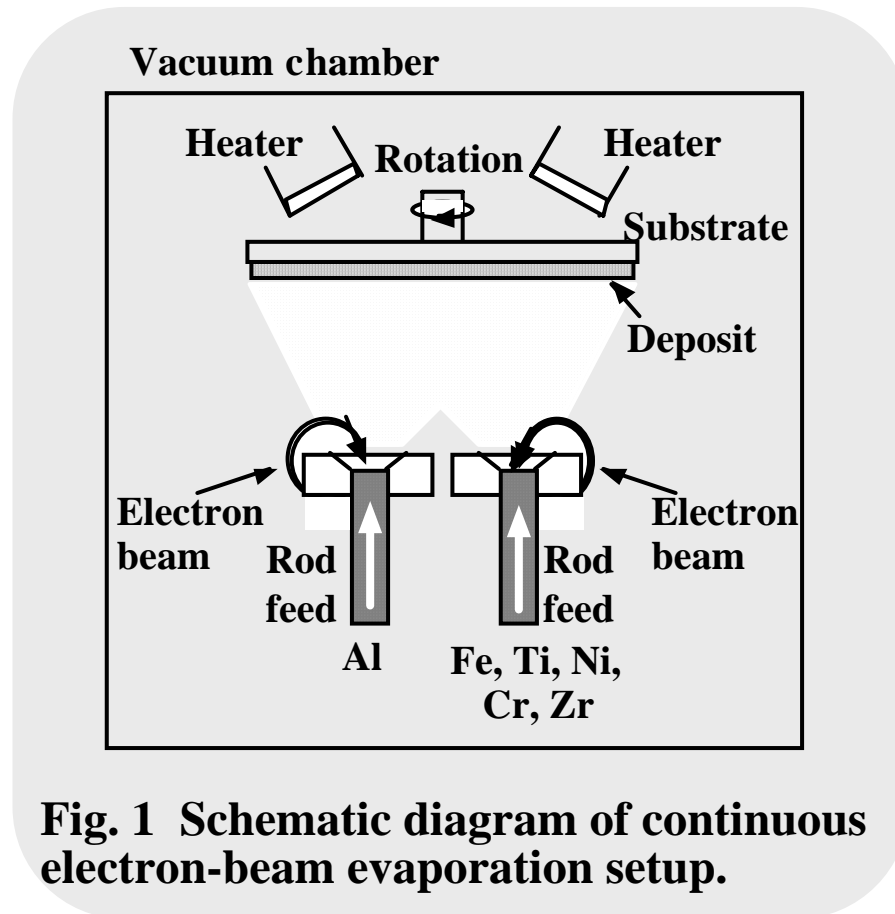
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- To investigate the strain rate dependence of mechanical properties in nano-crystalline metal alloy
- To know the fracture behavior of nano-structured alloy
- Development of high-strength Al alloy with sufficient ductility in a wide range of strain rate

# Proposed Processing

- Ultra-high speed quenching ( $\sim 10^{10}$  K/s) with Electron Beam Deposition - Vapor Quench(VQ) process.
- Composition controlled process with feedback
- Merit
  - Fabrication of super-saturated solid solution alloy

[Sasaki, Kita, Nagahora, Inoue:  
Mater. Trans, 42 (2001) 1561]



# Effect of Solute for Grain Refinement

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## Previous Trial

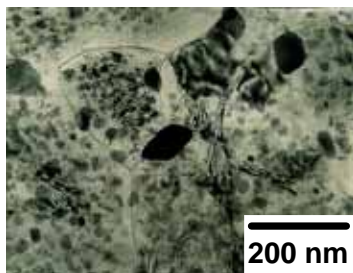
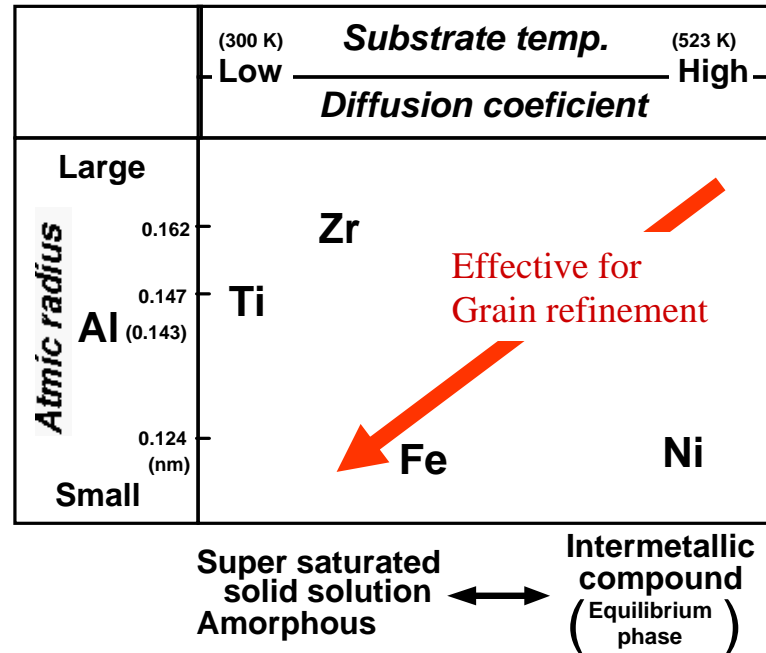
- Atomic radius
- Diffusion coefficient

Ni: Easy to form the  
Equilibrium phase

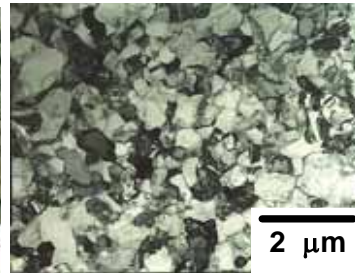
Ti: Easy to coarsening grains

Fe: Effective

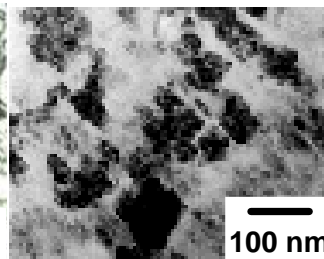
[Sasaki, Kita, Nagahora, (2001)]



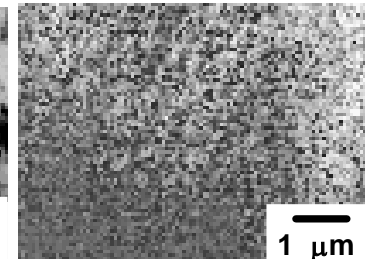
$\text{Al}_{99}\text{Ni}_1$



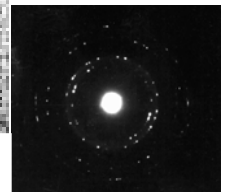
$\text{Al}_{87}\text{Ti}_{13}$



$\text{Al}_{98.5}\text{Fe}_{1.5}$



$\text{Al}_{90}\text{Fe}_{10}$

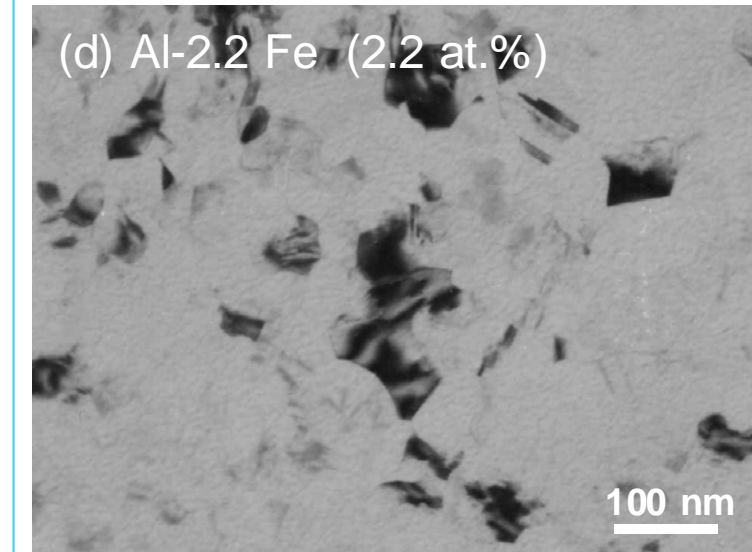
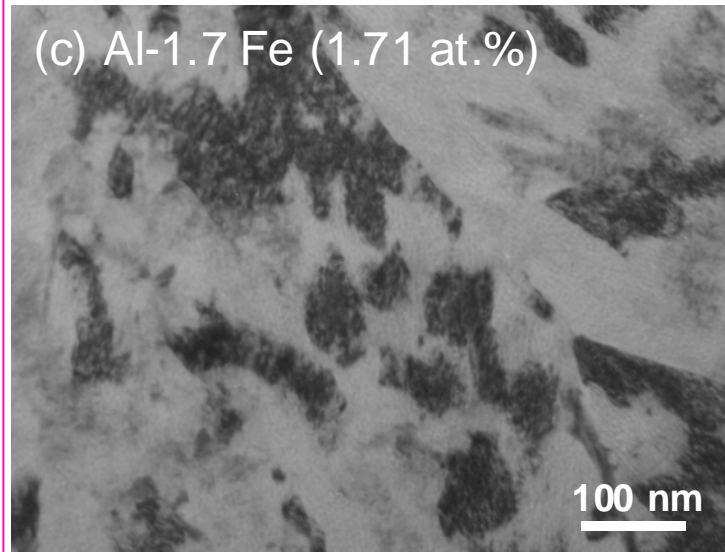
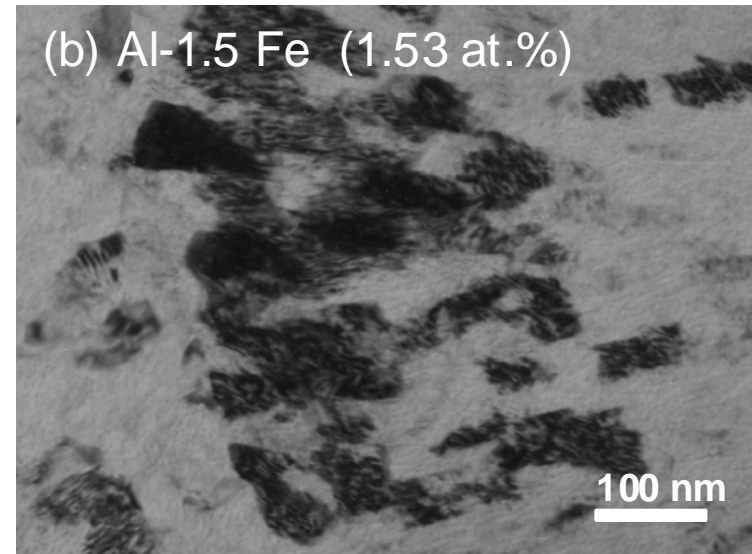
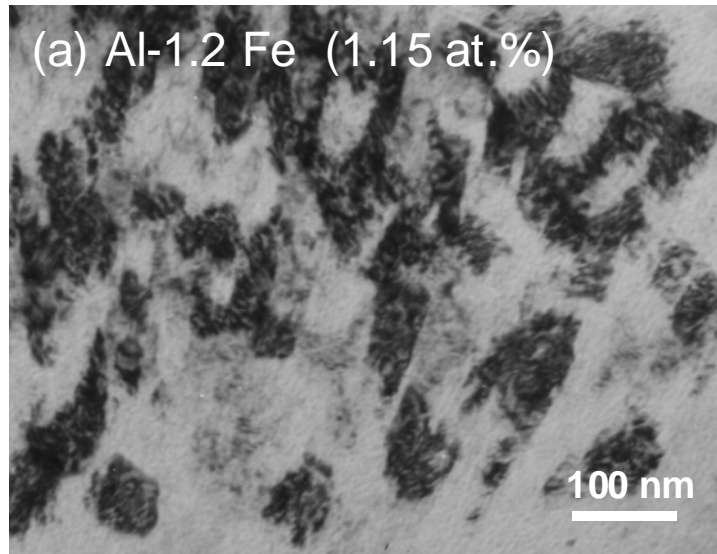




# Microstructural Characterization of Vapor-Quenched Al-Fe alloys

# Microstructure of VQ Al-Fe Alloy

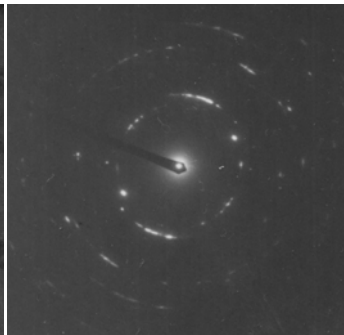
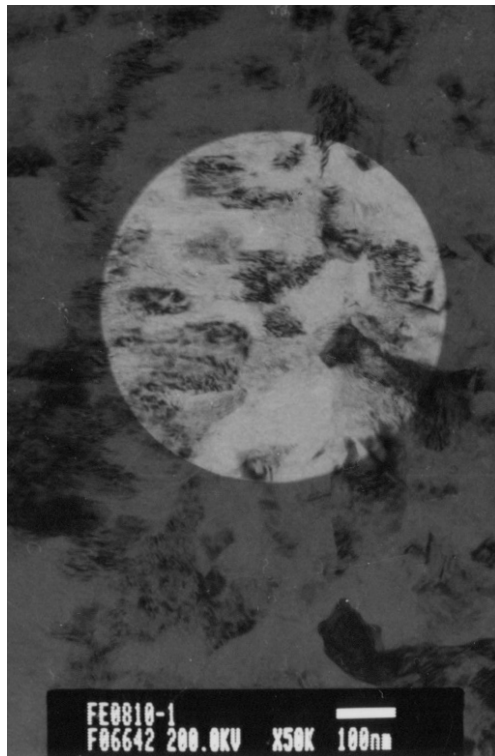
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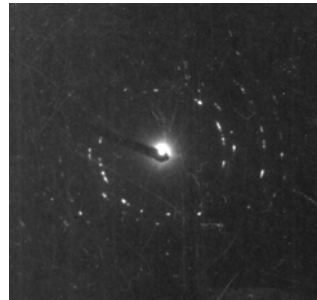
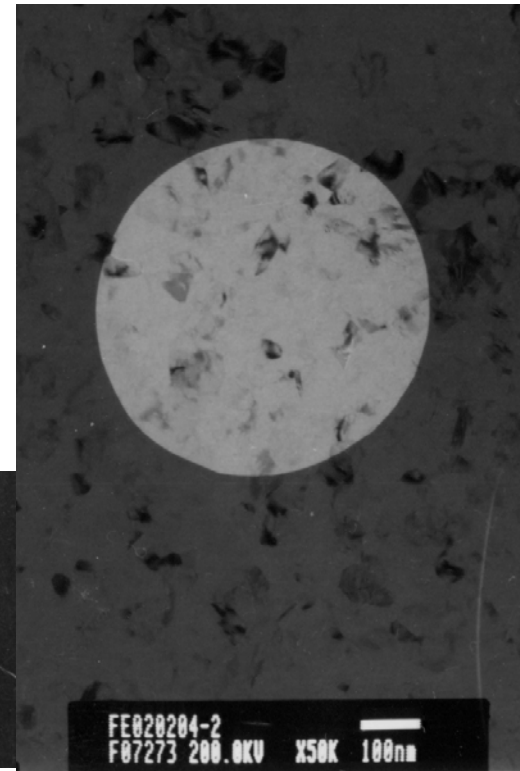
# Selected Area Diffraction Pattern of Al-Fe Alloy

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Al-1.5 Fe



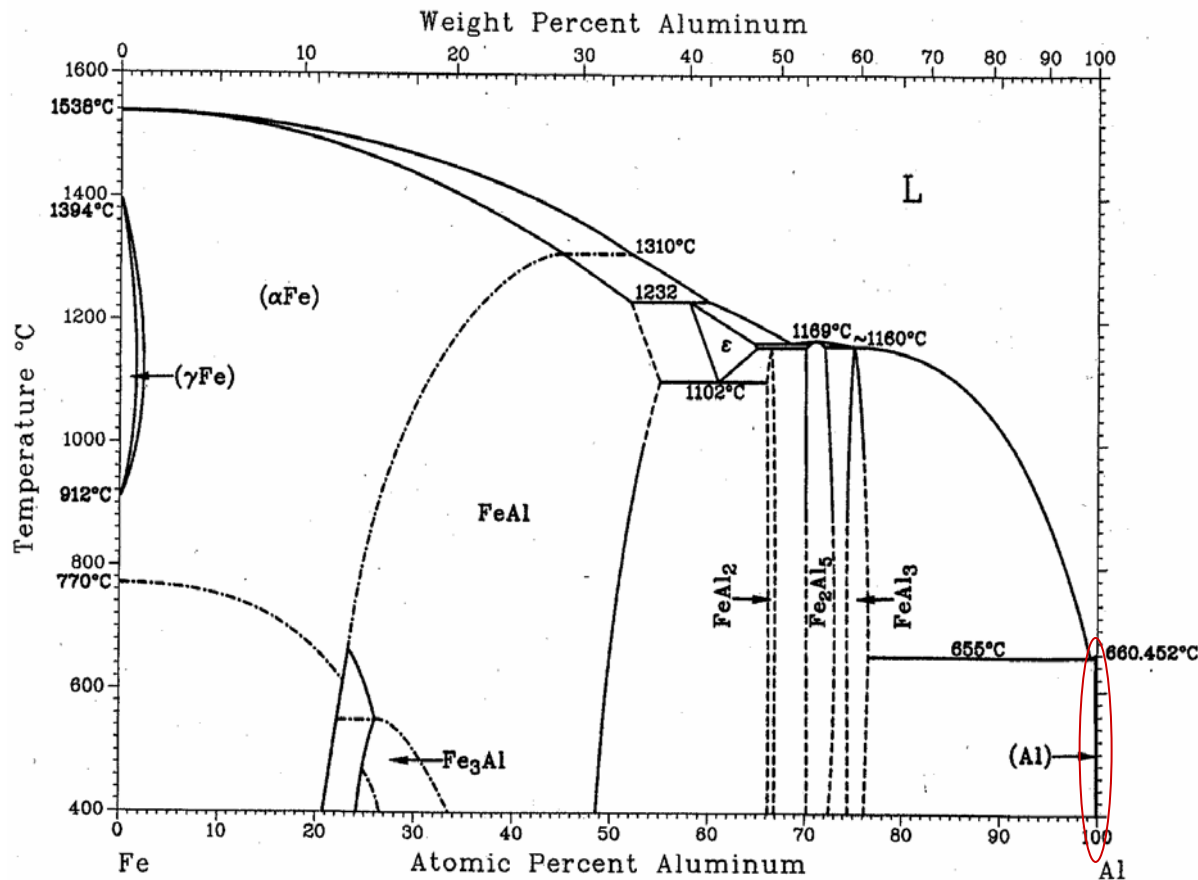
Al-2.2 Fe



- Distribution of grain orientation
  - Al-2.2 Fe alloy has larger distribution of orientation than Al-1.5 Fe alloy.

# Al-Fe Super-saturated Solid Solution Alloy

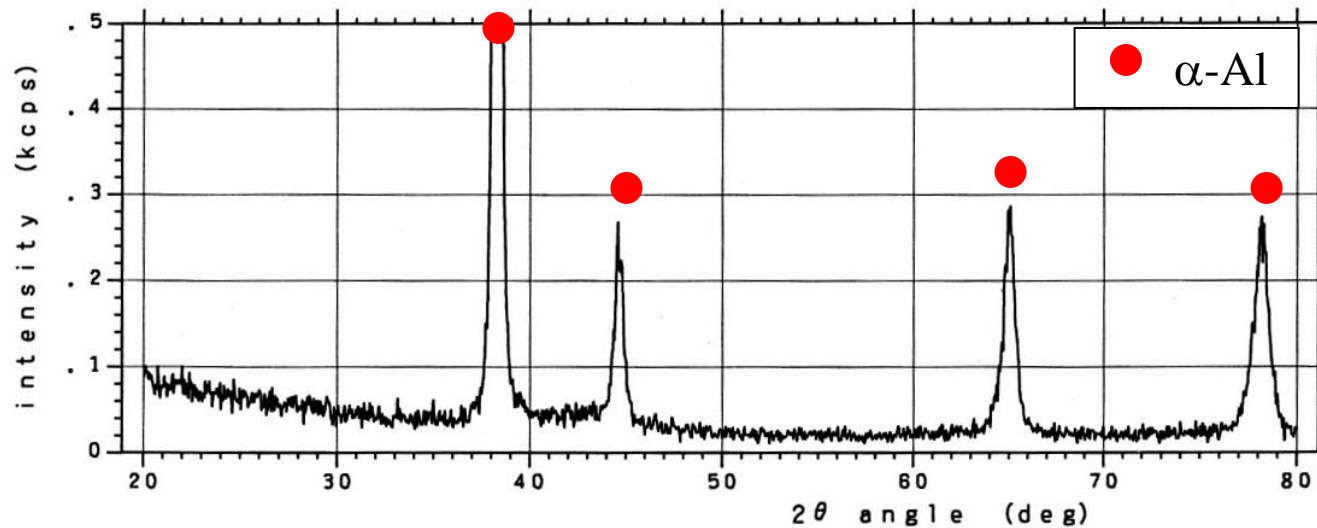
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- Al solid solution: solubility of up to 0.03 at.% Fe in Al
- Vapor quenching technique enables to fabricate super-saturated solid solution alloy up to 3 at.% Fe.

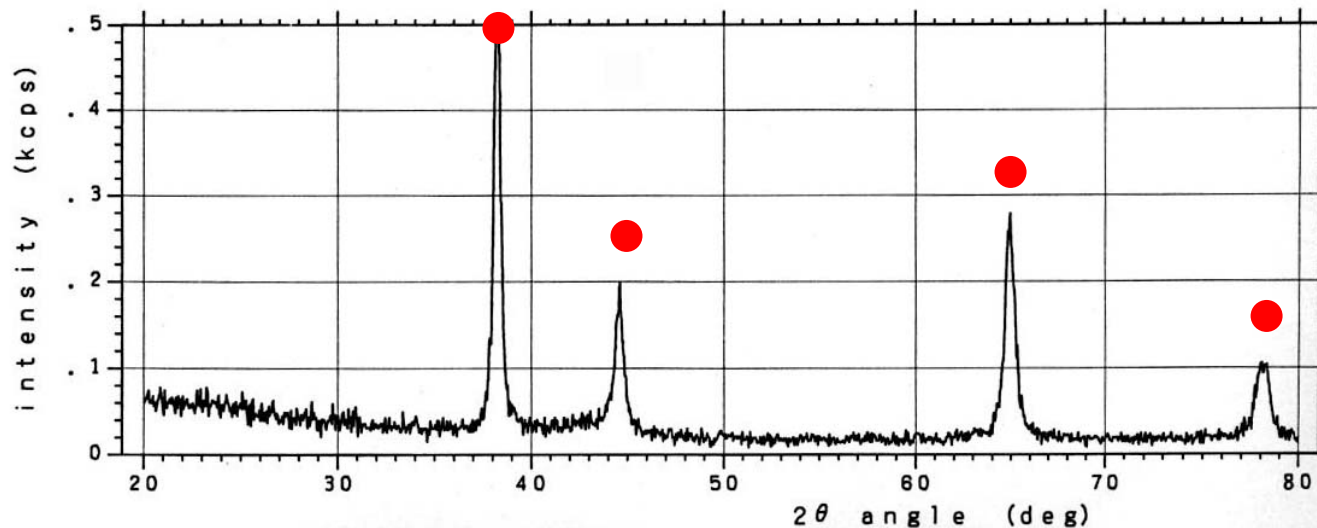
# X-ray Diffraction Pattern in VQ Al-Fe Alloys

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As deposited

Al-1.5 Fe



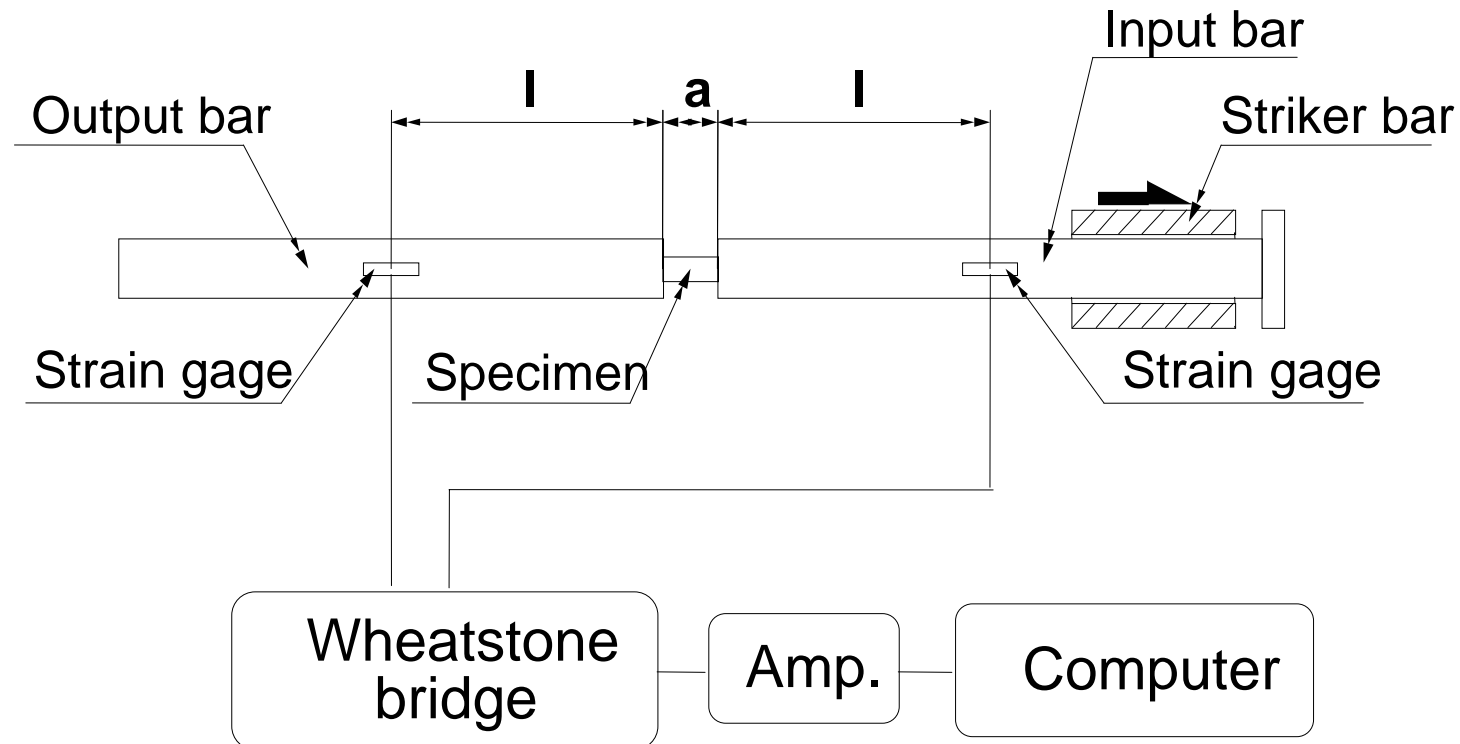
Al-2.2 Fe

- No second phase

# Mechanical Response of Nano-crystalline Al-Fe Alloys

# Tensile Tests

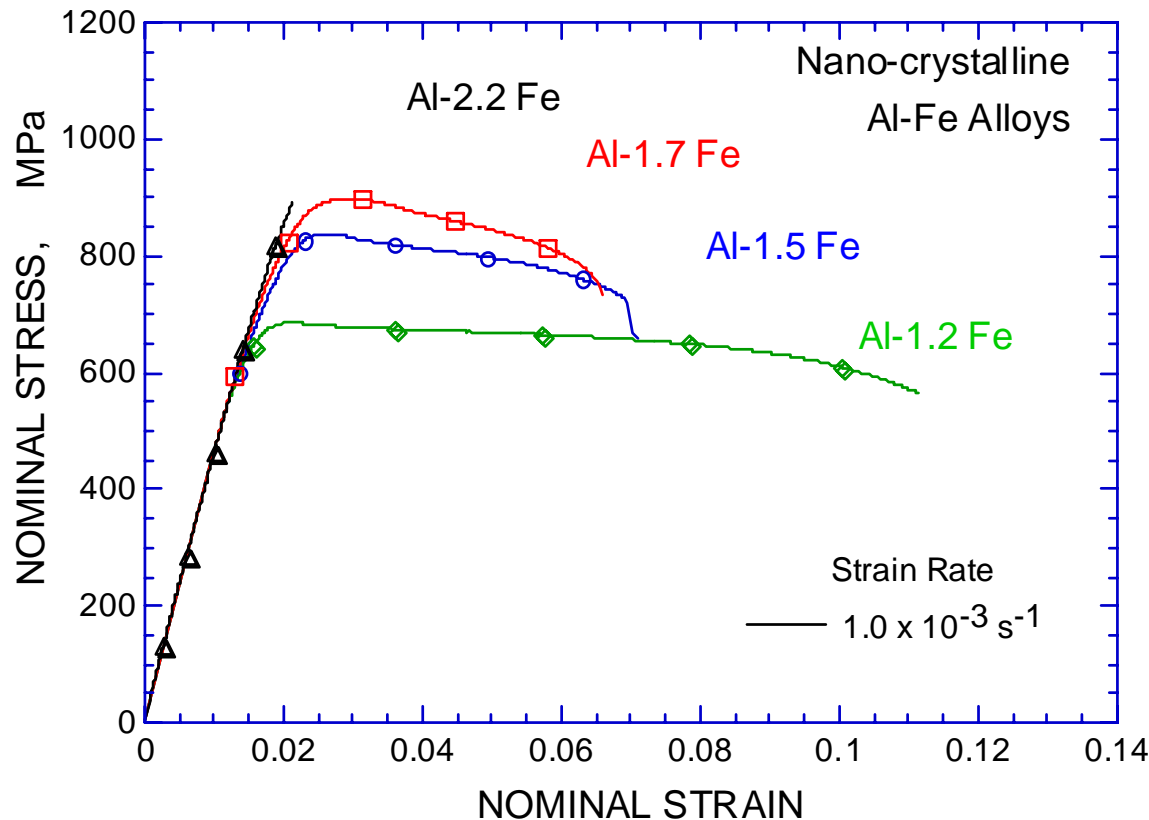
## Schematic Illustration of Tensile Hopkinson-bar



### Plate Specimen (Dog-bone type)

- Dimension - Gauge length: 9 mm, width: 3 mm  
Thickness: 0.6 mm
- Strain Rate :  $1100 \text{ s}^{-1}$  (Comparing with  $1 \times 10^{-3} \text{ s}^{-1}$ )

# Tensile Mechanical Response



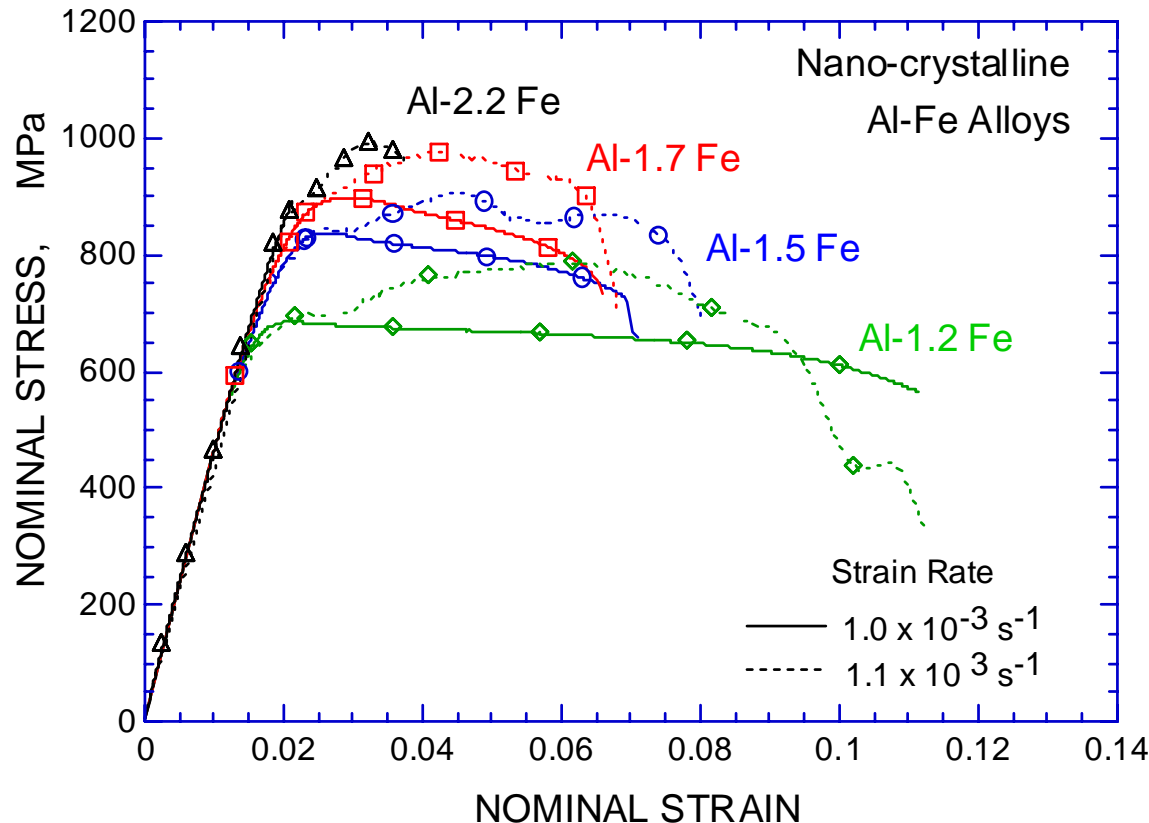
- Higher strength than conventional Al alloys.
- Strength increases and ductility decreases with increasing the content of Fe.
- Limited strain hardening



# Tensile Mechanical Response

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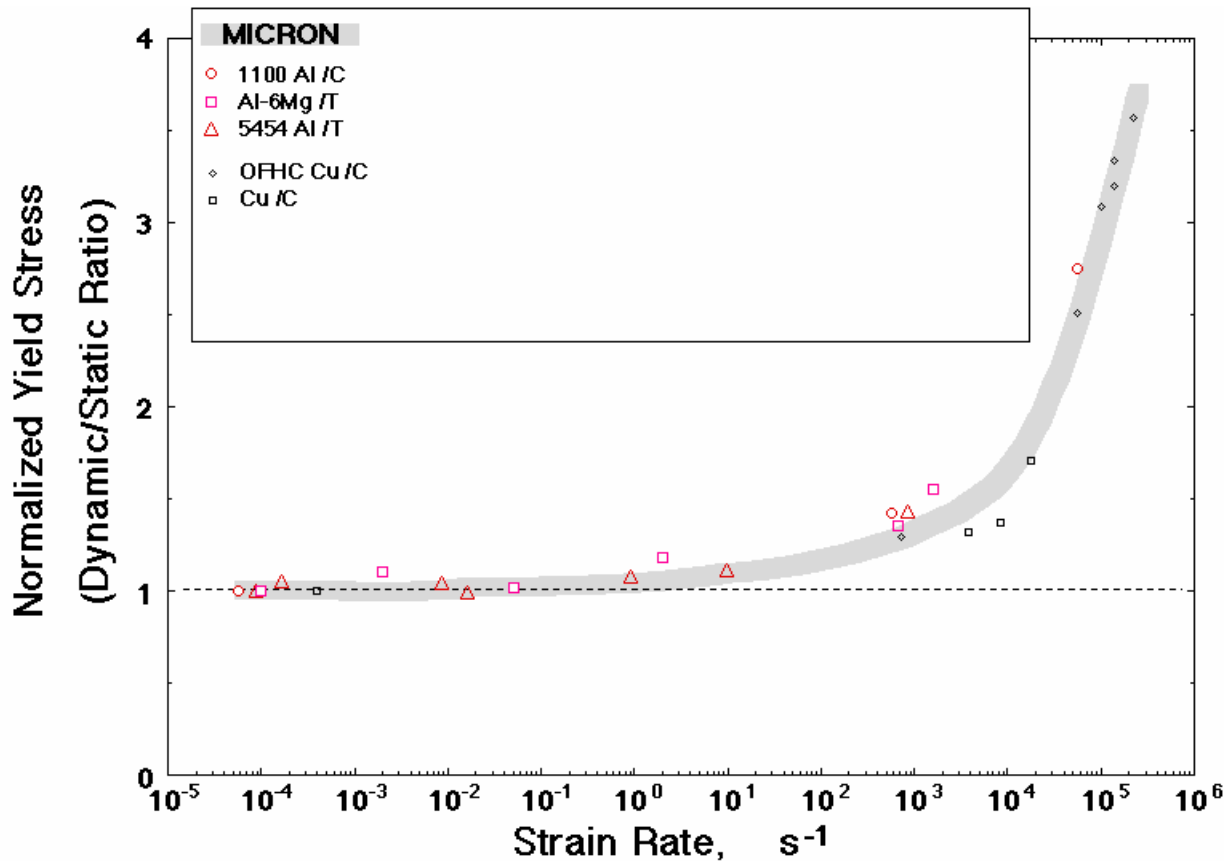
[Mukai, Suresh, Kita, Sasaki, Kobayashi, Higashi, Inoue: Acta Mater.,  
-in press]



- Al-1.7 Fe alloy exhibits a high strength  $\sim 950$  MPa with sufficient ductility more than 5% at a dynamic strain rate.
- Yield Stress is independent of strain rate, while flow stress and elongation-to-failure increase. The strain hardening rate is higher at the high strain rate.

# Strain Rate Dependence of Yield Stress

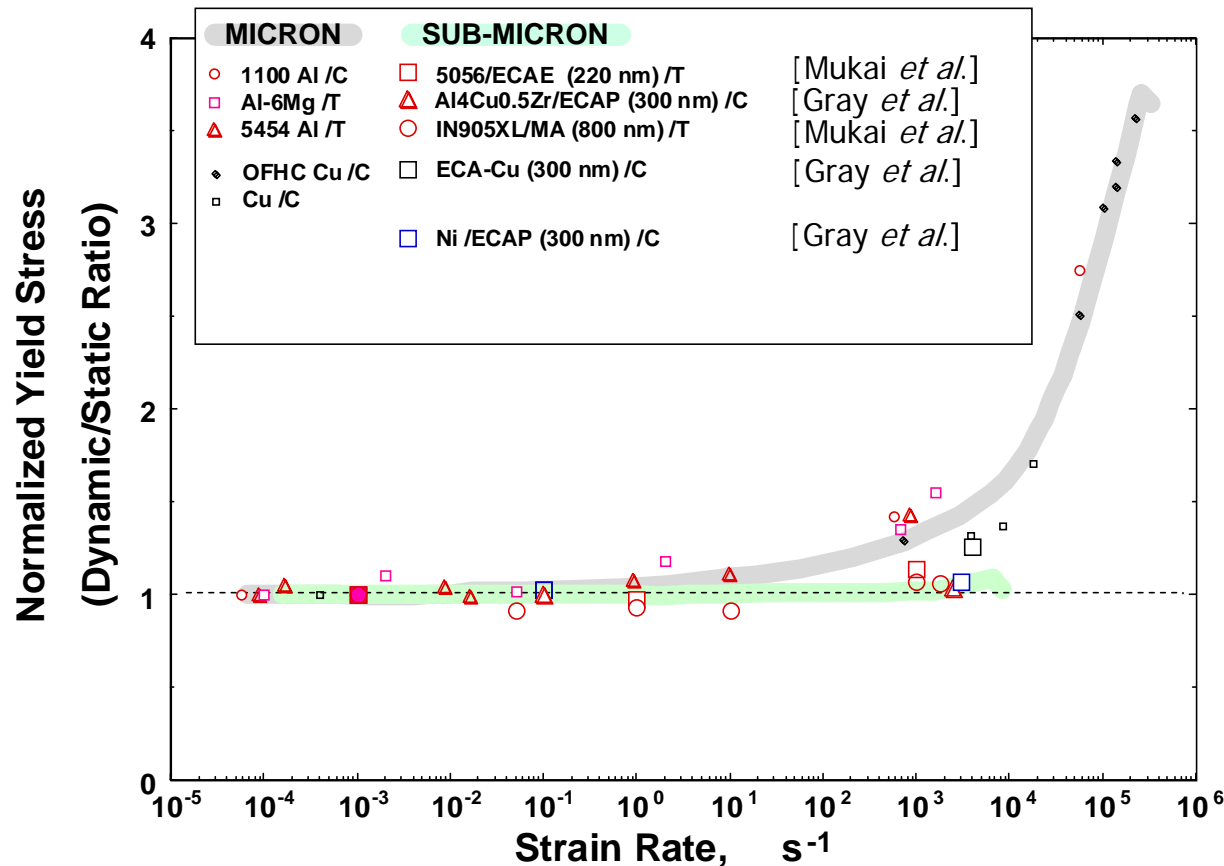
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- Micron crystalline alloy shows progressive strain rate sensitivity.

# Strain Rate Dependence of Yield Stress

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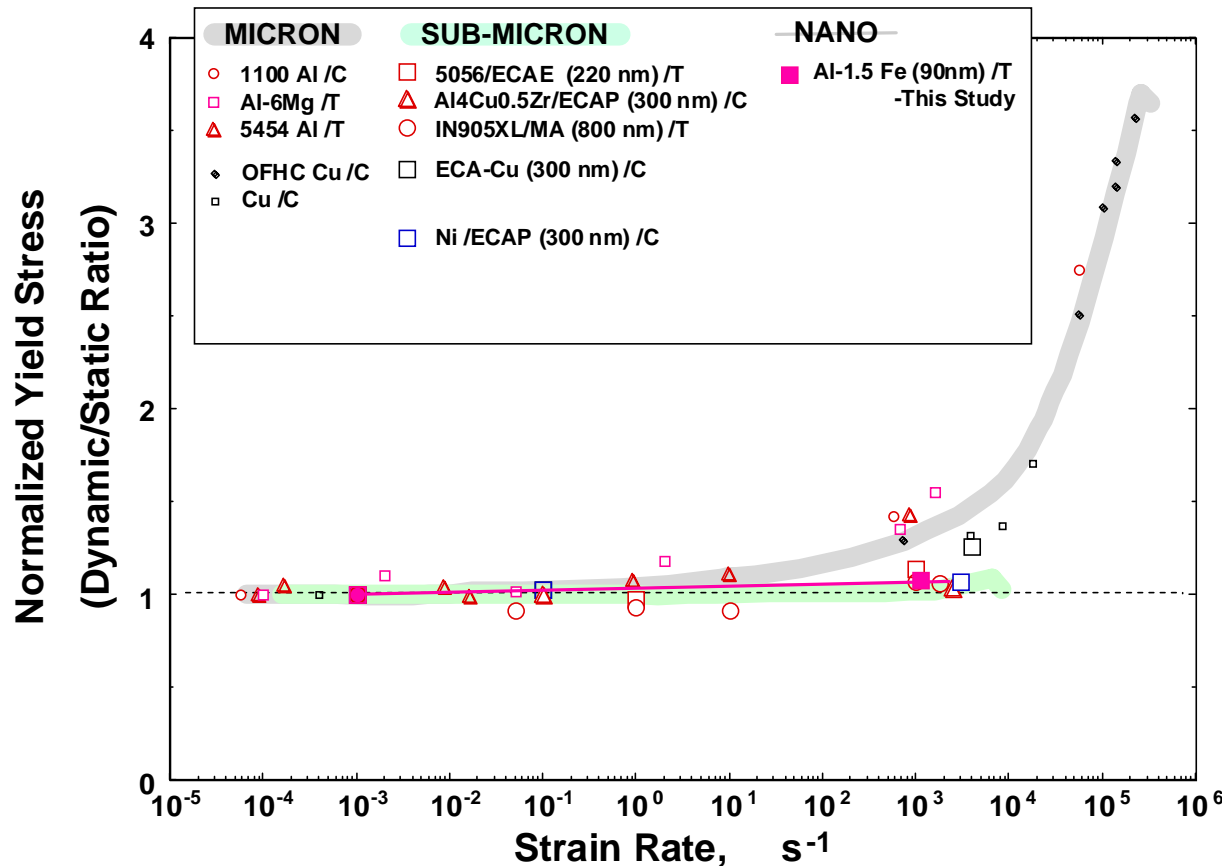
[Gray *et al.*, NanoStruct. Mater. (1997)]

[Mukai *et al.*, Met. Mater. Trans. (1995), NanoStruct. Mater. (1998)]

- Sub-micron-crystalline alloys exhibit low strain rate sensitivity.

# Strain Rate Dependence of Yield Stress

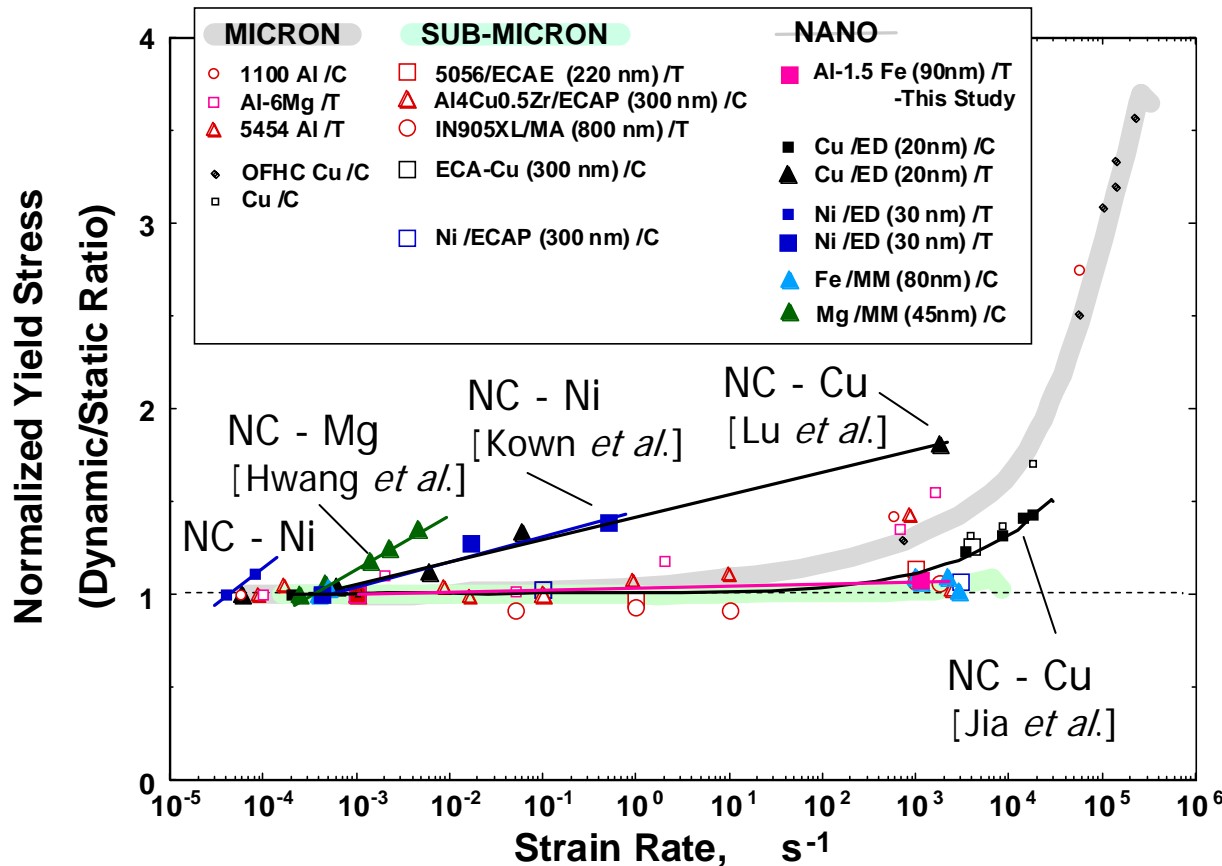
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- Nano-crystalline Al-Fe alloy exhibits low strain rate sensitivity
- For ECAP or MM alloys, highly constrained dislocation network limits the strain rate sensitivity. Another microstructural factor also limits the strain rate sensitivity of yield stress for NC Al-Fe alloys.

# Strain Rate Dependence of Yield Stress

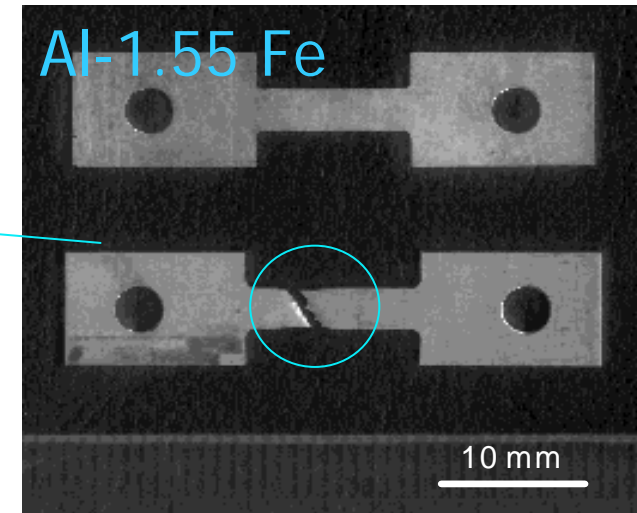
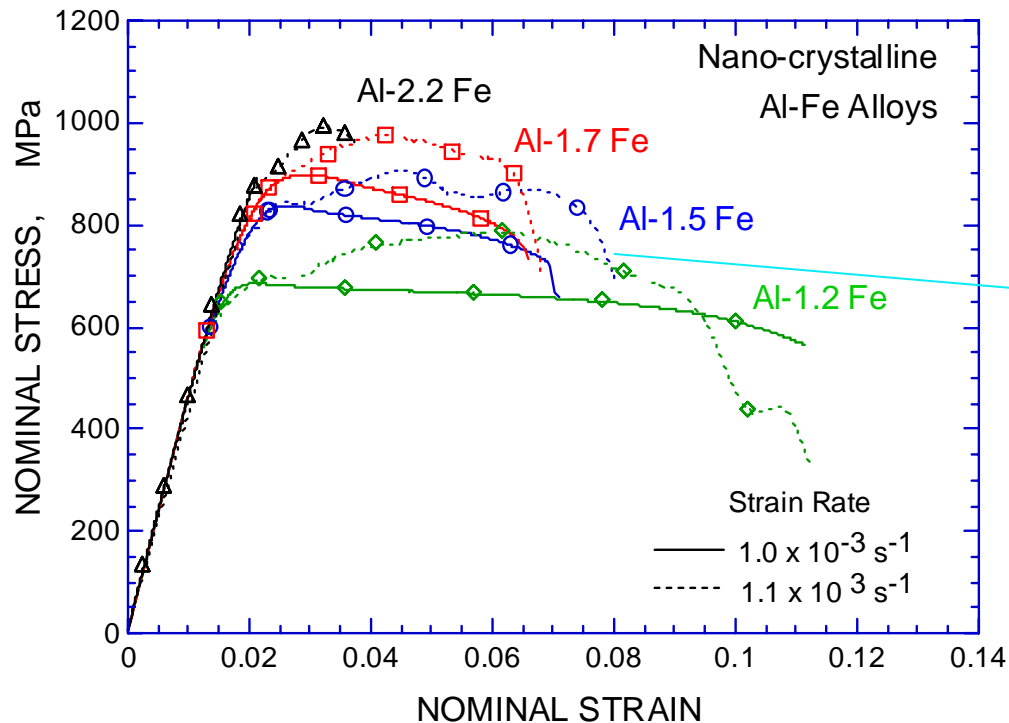
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- Some nano-crystalline pure metals exhibit strain rate sensitivity owing to the dominant deformation mechanisms, i.e., GBS or Coble creep.

# Tensile Mechanical Response

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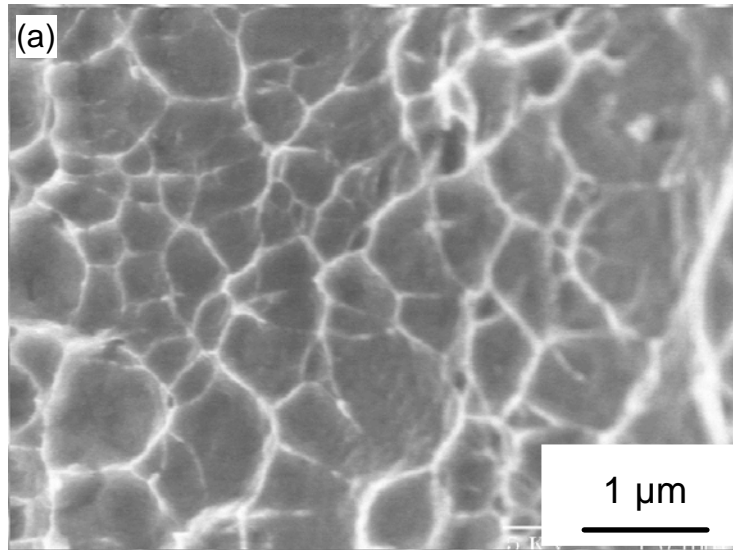


- Macroscopic fracture feature is noted to development of necking and formation of localized shear.

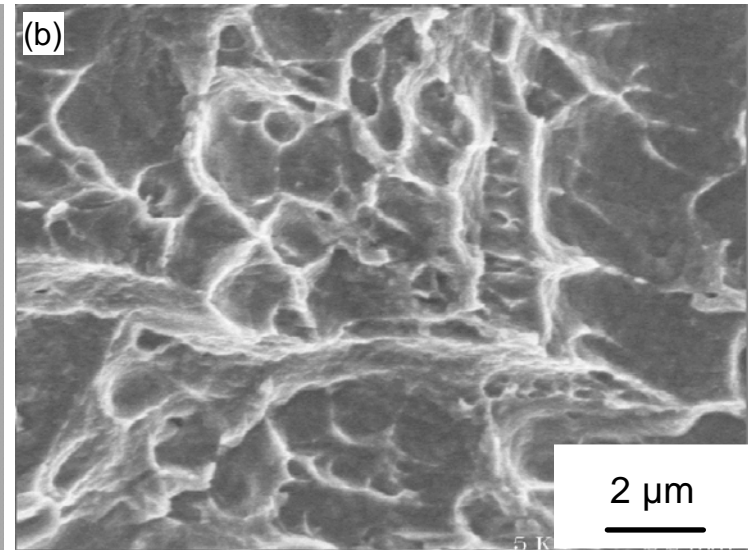
# Fracture Feature of VQ Al-Fe Alloys

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Univ. Tokyo

Al-1.7 Fe



Strain rate:  $1 \times 10^{-3} \text{ s}^{-1}$

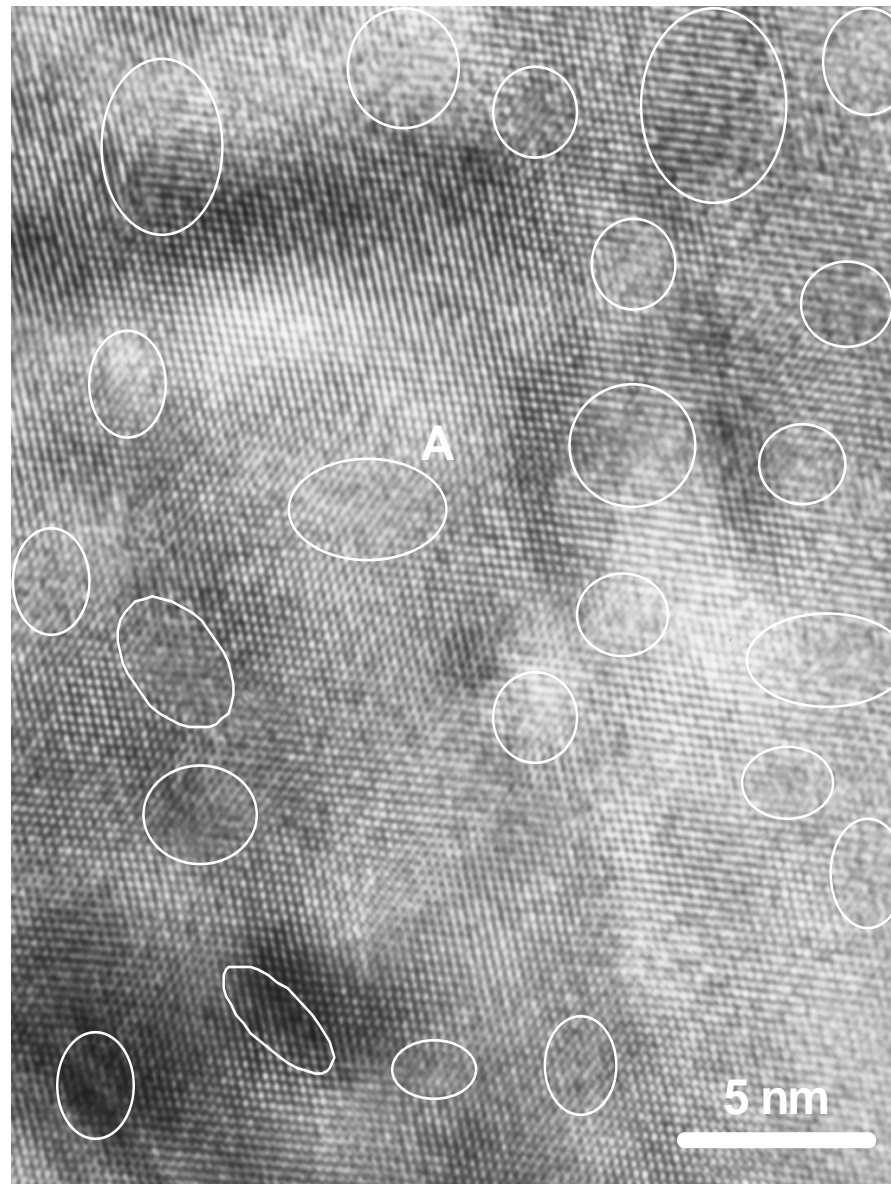


Strain rate:  $1.1 \times 10^3 \text{ s}^{-1}$

- Fracture surface typically consists of dimples.
  - >>> Evidence of dislocation motion interior of grains.
- Small and Shallow dimples at quasi-static strain rate suggest the limited ductility.

# Evidence of Uniformly Distributed Nano-Defects

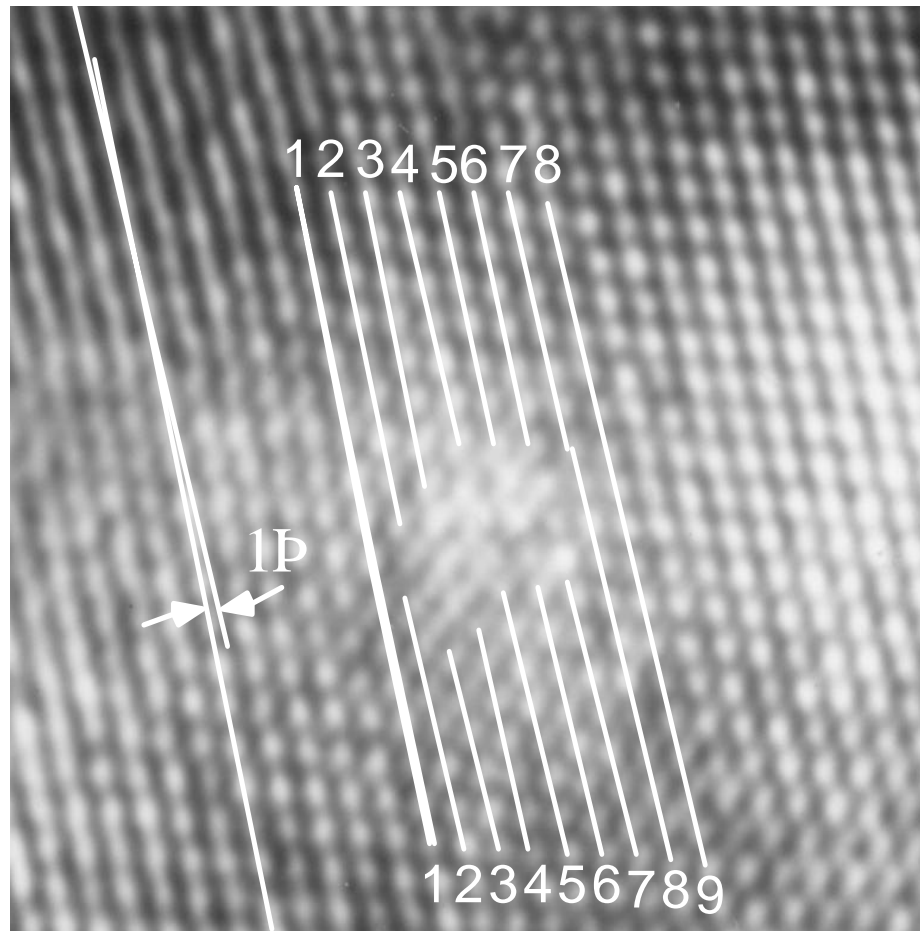
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*RCAST,*  
*Univ. Tokyo*





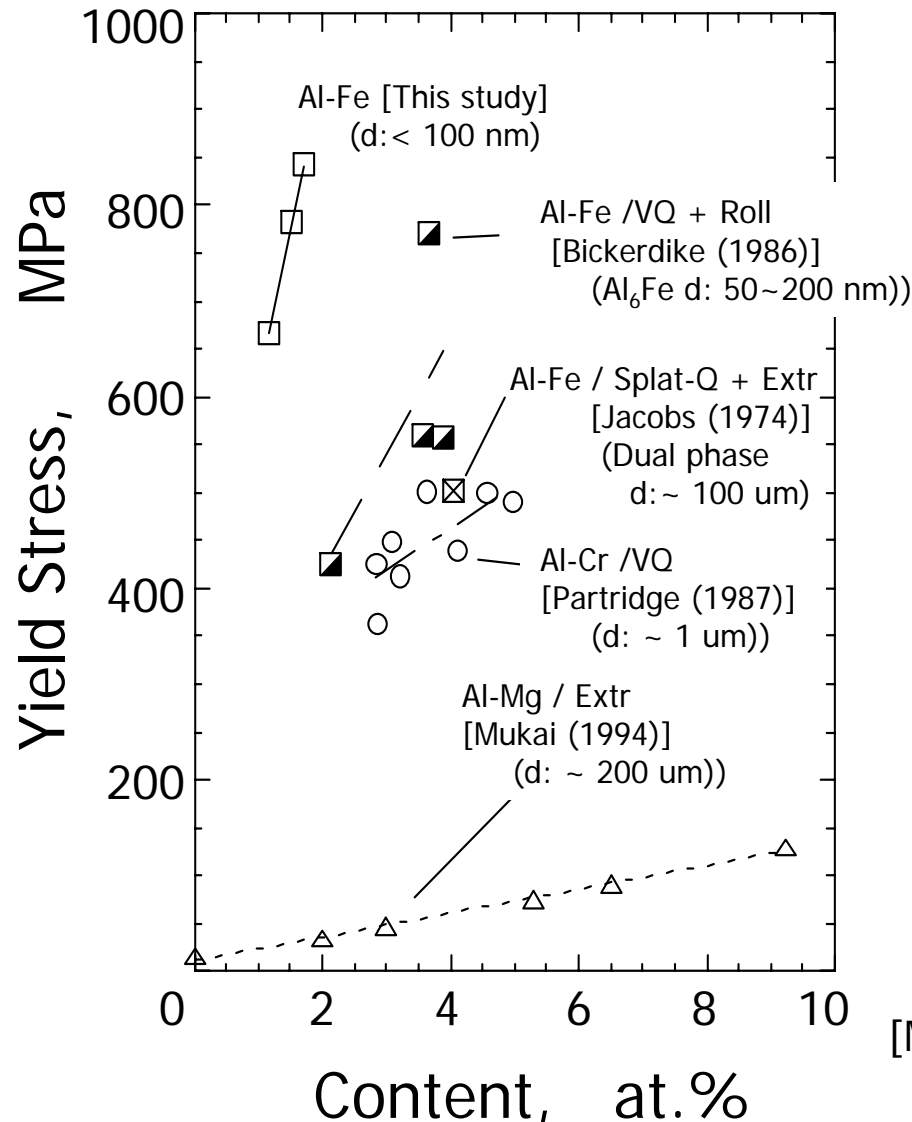
# Existence of Defect and Its Effect on Lattice Bending

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[Mukai, Suresh, Kita, Sasaki, Kobayashi, Higashi, Inoue: *Acta Mater.*,  
-in press]

# Influence of Concentration of Second Element on Strength in Binary Al Alloys



- Fe is effective element for strengthening.
- Strengthening rate is varied by its processing route.

[Mukai, Suresh, Kita, Sasaki, Kobayashi, Higashi, Inoue: Acta Mater., -in press]

# SUMMARY

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- Grain size of Al-Fe binary alloy was effectively refined with super-saturated iron solute. Formation of nano-crystalline structure was confirmed by TEM observation.
- Fine-grained Al-Fe alloys exhibit positive strain rate sensitivity of elongation-to-failure.
- The as-deposited Al-1.71 at.% Fe alloy showed an abnormal high tensile strength of  $\sim 950$  MPa and sufficient ductility of  $\sim 5$  % in tension at a high strain rate of  $1.1 \times 10^3 \text{ s}^{-1}$ .
- Fractography possibly suggests reduction of elongation in the nano-crystalline alloy with high angle grain boundary resulted in the intergranular fracture contrary to the the alloy with low angle grain boundary.